

## **COEI-2-METAUX Search for heavy metals**

### **1. Principle of the method**

Heavy metals react with the thiol function to form sulphurs. The coloration that results is compared to a standard.

### **2. Reagents**

- 2.1. Ammonium acetate,
- 2.2. Lead nitrate (II),
- 2.3. Glycerol,
- 2.4. Methanol,
- 2.5. Sodium hydroxide, solution at 1 mole NaOH /l,
- 2.6. Hydrochloric acid at 37%,
- 2.7. Thioacetamide reagent (R):
- 2.8. Standard lead solution:
  - 2.8.1. Lead solution at 1000 µg/ml: dissolve 1.598 g of lead nitrate(II) in water and complete to 1000 ml.
  - 2.8.2. Lead solution at 10 µg/ml. Add 10 ml of the solution 2.8.1 and complete to 1000 ml. To be prepared just before use.
- 2.9. Buffer solution, pH = 3.5: dissolve 6.25 g of ammonium acetate in 6 ml of water, add 6.4 ml of hydrochloric acid (2.6) and dilute with water until 25 ml.

### **3. Procedure**

- 3.1. Test solution: pour 5 ml of buffer solution (2.9), 25.0 g of sample and about 15 ml of water into a 50 ml graduated flask. Complete with water up to the reference mark.
- 3.2. Coloured solutions:
  - 3.2.1. Sample solution: mix 12.0 ml of test solution (3.1) and 2.0 ml of buffer solution (2.9) in a test tube.
  - 3.2.2. Comparative solution: mix 2.0 ml of test solution (3.1), 2.0 ml of buffer solution (2.9), 0.5 ml of standard lead solution (2.8.2), 4.5 ml of water and 5.0 ml of methanol in a test tube.
  - 3.2.3. Control solution: mix 12.0 ml of test solution (3.1), 2.0 ml of buffer solution (2.9) and 0.5 ml of standard lead solution (2.8.2) in a test tube.
  - 3.2.4. Comparison of colorations:

# INTERNATIONAL OENOLOGICAL CODEX

## METAUX LOURDS - RECHERCHE

---

- add 1.2 ml of thioacetamide reagent (2.7) in the 3 test tubes (3.2.1 to 3), mix and wait 2 minutes. Compare the coloration vertically in the light of day.
- the sample solution must not be darker than the comparative solution.
- the control solution must not be lighter than the comparative solution.

#### 4. **Results:**

The conditions described in 3.2.4 are obtained if the heavy metal content is less than 10 mg/l expressed in lead and with a precision of 1 mg/l.